New Manufacturing Process Found for High-Performance Polyimides

A new chemical technique for producing high-performance polyimide materials may make the widely-used polymers easier to process. Polyimides are used extensively in electronics manufacture and high-performance aerospace composites. Their high strength, ability to withstand high temperatures, and immunity to moisture make the materials valuable, but also difficult to process, especially in coating applications.

"The factors that make the polyimides desirable as materials also make them very difficult to process," said Laren M. Tolbert, professor of chemistry at the Georgia Institute of Technology. "They are very good vapor barriers and they are very tough polymers. These factors also mean they are insoluble and don't flow or process well."

The method Tolbert and colleague Zhanqi He are studying, known as Diels-Alder polymerization, joins short chains—oligomers—in the precursors to produce strong polyimide chains in such a way that the links are indistinguishable from the chain. This method prevents production of water vapor in subsequent curing, a previously encountered problem that can leave pinholes, compromising the protection offered by a polyimide coating.

In use, the precursor materials would be mixed together, then spread as a coating on an electronic component or laid up as part of a composite structure. The coating would then be heated to induce curing, or subjected to the normal consolidation process used for composite components.

"The main advantage is that this technique provides a way of curing polyimides without having to use the pre-polymer," Tolbert said. "You could cure a resin that would contain all of the components you needed. You could also cure a layered structure by putting it into the press and heating it to cause the reaction to occur, without worrying about the evolution of water."

This process produces polyimides more expensively; however, preliminary testing shows that the resulting material possesses thermal stability comparable to other polyimides.

Sandia and Radiant Technologies to Develop Improved Chip Memory

Sandia National Laboratories and Radiant Technologies Inc., of Albuquerque, have signed a cooperative research and development agreement (CRADA) to develop a new type of nondestructive readout (NDRO) nonvolatile semiconductor memory device. Ferroelectric thin films will provide the nonvolatile property. This kind of memory holds significance for optical communication systems and military computers, and may help integrate diverse technologies into commercial products.

Nonvolatile memory, which retains all information even after a loss of electrical power, is replacing traditional memory elements in many microelectronic and optoelectronic systems. One problem with such memories is that every time they are read, they have to be rewritten to their original state by a brief "destructive readout" that takes about 0.1 μs. During that instant, if something else happens, such as a single-event upset, the information can be permanently scrambled. A method of nondestructive readout would avert that.

Under the CRADA, Sandia and Radiant Technologies will develop an NDRO nonvolatile memory device by adding a semiconductor thin film above the ferroelectric thin film layer. The ferroelectric material used by the researchers is lead zirconate titanate (PZT). In the proposed device, the conductivity of the semiconductor thin film is modulated by the polarization state of the underlying PZT film layer. Monitoring the current thus provides a direct nondestructive readout of the stored data.

The three-year agreement calls for Radiant Technologies to coordinate the program, design and manufacture the prototype NDRO nonvolatile semiconductor memory, and help design plasma-enhanced metallo-organic chemical vapor deposition equipment that will be used to make the thin films. Sandia will characterize the PZT-semiconductor thin film interface and investigate methods for measuring film properties and for determining interfacial reactions. Sandia will also contract with researchers at the New Mexico Institute of Mining and Technology in Socorro for characterizing fatigue in ferroelectric thin films.

In addition to nonvolatile semiconductor memories, the films can be used in high-speed optical switches and modulators, 3-D holographic TV systems, image comparators, optical disk storage, infrared detectors, and sensors for light, chemicals, motion, sound, and pressure.

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Materials Science Advancements in Japan

Laser Produces High T_c Superconductor

Osaka University researchers have succeeded in producing a superconductor, structured exactly as they had designed it, by building up atoms layer-by-layer. Conventional ways of producing a superconductive substance depend on chance: materials are ground, kneaded, and baked just as they would be in making ceramic ware. Thus, the atomic structure that produces superconductivity is formed by chance.

In the new method, used by Tomoji Kawai and Shichio Kawai, laser beams are used to realign the atoms of a material, which is built up epitaxially. Using this method, the researchers produced a substance consisting of layers alternating between calcium-strontium and copper oxide. In the experiment, the product turned superconductive at temperatures between 130 and 120 K, the highest temperature achieved for superconductivity. The thin films are synthesized at ambient pressure with 10^{-5} torr of NO_2. A Kyoto University team is known to have produced the same substance, achieving a T_c of 100 K, but in the conventional way, using a powder and high pressure ($\sim$6GPa).

Carbon Nanotubes Produced in Bulk

Researchers at NEC Corporation report that they have found a way of producing a new type of carbon material in quantities large enough to test its properties. The carbon material, called carbon nanotubes, consists of cylinders made of hexagonal lattices of carbon atoms—like a tube made from chicken wire.

The NEC researchers, Thomas W. Ebbesen and P.M. Ajayan, first discovered how to make the nanotubes last March while trying to make derivatives of fullerenes. Their technique—announced in the July 16 issue of *Nature*—simply and consistently produces gram quantities of carbon nanotubes.

As in forming fullerenes, the technique involves passing an electric current between two graphite electrodes in an inert atmosphere, preferably helium. One of the electrodes is consumed and a cylindrical deposit forms on the other electrode. The deposit has an outer hard shell without nanotubes and a soft fibrous core made of nothing but nanotubes and nanoparticles of carbon. The key to producing a higher yield of nanotubes is the pressure of the gas in the reaction chamber. At about 500 torr helium, the yield of nanotubes reaches 25% of the weight of the initial graphite electrode, comparing favorably to the yield of fullerenes, which is only about 1%.

The nanotubes are micrometers long but only two to 20 nanometers in diameter. With such a large aspect ratio and nearly perfect crystalline structure, carbon nanotubes should be very useful for strengthening other materials by making composites. Another intriguing feature of the nanotubes is that they come with varying degrees of helicity and in various diameters, which theoretical calculations predict will lead to a variety of electronic properties. "The tube acts either like a metal or a semiconductor depending on the degree of helicity and its diameter," said Hitoshi Igarashi, a senior manager at NEC's Fundamental Research Laboratories. Although mostly bulk properties have been measured to date, efforts are under way to study single tube properties.

Manufacturing Method Controls Microstructure of Silicon Nitride

The National Institute for Research in Inorganic Materials (NIRIM) of the Science and Technology Agency and the Central Research Institute of Nissan Motor Co. Ltd. have jointly developed a new method to manufacture high-strength and high-reliability silicon nitride ceramics to be used as automobile engine parts, precise machine parts, and cutting tools. The fundamental principle for the improvement of mechanical properties was developed at NIRIM and the materials are called "*in-situ* composites." The researchers grew large rodlike grains in fine matrix grains during gas-pressure sintering to give a "composite" microstructure.

Silicon nitride ceramics have high strength at high temperatures, high thermal shock resistance, and other desirable properties for engineering ceramics. For industrial applications, however, they have low reliability (wide strength distribution) and high cost compared to metallic parts. The most important requirements for the fabrication of high-strength and tough ceramics using conventional sintering methods is to use high-purity (chemically and crystallographically) alpha powders with fine and homogeneous grain size. The mixture of silicon nitride and oxide additive reacts to form a liquid phase at high temperatures. The alpha to beta phase transformation takes place during liquid-phase sintering. Some beta grains grow irregularly into fine matrix grains during the sintering. The microstructures of *in-situ* composites, therefore, cannot be controlled in conventional methods, thus leading to a wide distribution of strengths.

The new method uses beta powder, which gives a homogeneous microstructure after sintering. A high sintering temperature greater than 1900°C is employed in gas-pressure sintering. The grain size distribution in the beta powder is large by design, which accelerates the growth of large and rodlike grains from nuclei. The size and distribution of the rodlike grains is easily controlled in this method. The distribution of strength values is very narrow, less than 3%, because of the controlled microstructure. The microstructure is also controlled to give high fracture toughness. The method is applicable to the sintering of low-cost powder which is composed of mainly beta form.

300 Å Superconducting Thin Films Produced by NRIM

The Tsukuba Laboratories of the National Research Institute for Metals (NRIM) of the Science and Technology Agency recently produced a 300 Å bismuth-oxide, high-temperature, superconducting film. In tests, NRIM attained a superconductive critical temperature of 108 K, the highest temperature achieved for a bismuth-oxide thin film.

High-quality thin films of high-temperature superconducting oxides are needed if high-temperature superconductors are to be used in electronic devices. Development of an extremely thin material with excellent electrical properties, surface flatness, homogeneity, and structural stability is a difficult task. Thin films of this kind have been manufactured using vacuum deposition, electron-beam deposition, chemical vapor deposition, and sputter deposition. When the films became thinner than about 1000 Å, however, critical temperatures exceeding 100 K could not be attained.

By adding an ion irradiation step, followed by heating, the NRIM researchers produced films as thin as 200-250 Å with T_c still above 100 K. With a magnetron sputtering method using bismuth-strontium-calcium-copper-oxide powder as a target, the institute manufactured thin films of excellent quality and a thickness of 300 Å or less on the magnesium oxide substrate. After heat processing, the material was irradiated with inert gas ions and heated a second time to rearrange the imperfect crystal into a more perfect one. A critical temperature of 108 K was achieved.

F.S. Myers

S.K. Wolterman Named Young Engineer of the Year

Sandra K. Wolterman has been named 1992 Young Engineer of the Year by the New York State Society of Professional Engineers during the Society's Annual Meeting in Rochester, New York. Wolterman received the honor for her educational and engineering achievements; professional society, civic, and humanitarian activities; demonstrated competence; and contributions to the goals of the Society of Professional Engineers. The award honors professional engineers under the age of 35.

Currently pursuing a PhD in Materials Engineering from Rensselaer Polytechnic Institute (RPI), Wolterman is a licensed professional engineer in New York State. She received her bachelor's degree in industrial engineering from the University of Iowa and a master's degree in mechanical engineering from RPI. Wolterman is manager of semiconductor measurement and inspection equipment engineering at IBM Corporation.

E-MRS Holds Elections

During the 1992 E-MRS Spring Meeting, elections took place within the Executive Committee of the European Materials Research Society. Paul Siffert (CRN, Strasbourg) will remain as general secretary, and Ian W. Boyd (University College London) will continue serving as second vice president.

P.A. Glasow (Siemens, Germany) will be the next president of E-MRS, replacing current president Bernd Stritzker (IFK, Julich). J.G. Wurm (consultant and former division head with the European Communities) will be vice president.

The executive committee acknowleged the new officers' extensive experience with industry and European research. Successful maturation of E-MRS, said the committee, will depend not only on the continued efforts of well-established leaders, but also on the influx of new people and ideas. It was therefore recommended that the 1993 call for candidates for elections to the Board of Delegates be widely circulated to attract energetic, young scientists across Europe.

A.N. Cormack Appointed Dean at Alfred

Alastair N. Cormack, professor and associate dean for Graduate Programs at the New York State School of Ceramic Engineering and Sciences at Alfred University, has been appointed dean of the School. Cormack, a member of the faculty for the past seven years, said his first goal would be to strengthen the school's already strong ceramics program.

"Research activities are an inherent part of the school," he said. "I would like to further develop our research programs, which are so integral to graduate student and faculty development." Cormack also said that while the School's faculty consists

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of well-known researchers, they are still dedicated to teaching, especially to teaching undergraduates.

His research interest is in the theoretical treatment of complex defect behavior in noncrystalline and crystalline ceramics, using computer simulations of atomic structure.

Cormack came to Alfred in 1985, and holds a doctoral degree from the University College of Wales, Aberystwyth.

Modeling of Microscopic Crashes Reveals Environments for New Chemistry

Driving clusters of atoms or molecules against solid surfaces can generate forces that produce new chemical environments and promote new reaction processes, according to Georgia Institute of Technology researchers in the July 17 issue of *Science.* Uzi Landman and Charles Cleveland, using supercomputer simulations at Georgia Tech's School of Physics, found that the supersonic collisions cause extreme pressure, density, and temperature pulses. "We have very little knowledge about chemistry under these extreme conditions," Landman said. "Compounds that may not form under normal conditions may be produced under these circumstances."

Clusters moving at 2-5 km/s create pressures of up to 100,000 atm and temperatures as high as 4000°C when they hit a solid surface. Modeling done by the researchers shows that the volume of normally incompressible materials could be reduced in this environment. Such conditions last in the picosecond range during which atoms are moved much closer together than normal and their ability to share energy is greatly reduced. Landman believes the collision of clusters could act to catalyze and promote reactions otherwise difficult or impossible to generate.

As atoms in the leading edge of the cluster contact the solid surface, their velocities drop precipitously. The atoms following them, however, are still moving at high speed, causing an atomic "pileup" as they crash into the front edge of the cluster. The pileup in the inertially confined medium creates a shock wave, which for a few picoseconds compresses the cluster. That compression unleashes tremendous energy able to break chemical bonds and alter other atomic characteristics. After the impact, shock pulses also move in a direction opposite to the shock wave traveling through the solid. That causes portions of the cluster to explode outward and laterally, ultimately spreading across the surface of the solid. Approximately three-quarters of the energy of the impact is absorbed by the substrate, creating shock waves that can physically alter the surface of the material.

The researchers studied the effects of crashing argon clusters into a surface of sodium chloride and sodium fluoride. Since submission of the *Science* paper, Landman, Cleveland, and H.P. Cheng—also in the School of Physics—have been exploring how the microscopic crashes will affect tiny crystals of sodium chloride and sodium fluoride. The research suggests that a new type of fragmentation or fracture may occur, opening yet another set of research

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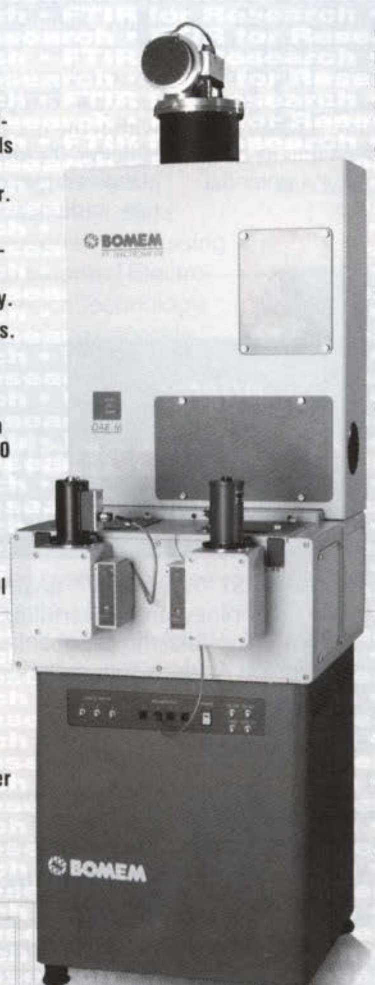

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Penn State Researchers Show That Synthetic Mica Can Capture, Store Strontium 90

Researchers at Pennsylvania State University's Materials Research Laboratory have manufactured a substance, Na-4-mica, that seizes strontium 90 (^{90}Sr) in aqueous solutions and locks it into its own structure, effectively removing a potential radioactive hazard from water.

Natural mica contains aluminum, silica, magnesium, and potassium, while the synthesized Na-4-mica has two sodium ions in place of each potassium ion. The researchers—Sridhar Komarneni, graduate student William J. Paulus (now with General Motors), and Rustum Roy—found the sodium substitution caused an offsetting of the inherent layering of the mica structure, allowing water molecules to enter it. As this happens, Na-4-mica preferentially exchanges two sodium ions for any available ^{90}Sr ions.

Komarneni, professor of clay mineralogy in the Materials Research Laboratory and the College of Agricultural Sciences at Penn State, said, "When about half the spaces have been filled by ^{90}Sr ions, the layer realigns and the offset disappears, effectively trapping the ^{90}Sr permanently within the mica's structure." Mica, he said, is better for storing radioactive ions than glass—a reference to a method used to contain radioactive cesium removed from waste water at the Three Mile Island nuclear energy plant. According to Komarneni, mica is even more stable and less affected by chemical weathering than glass.

In the June 18 journal *Nature*, the researchers reported manufacturing significant amounts of Na-4-mica. "We developed a technique to synthesize large quantities of Na-4-mica with very small particle size, which is very important because it provides a large surface area for the reaction to take place," Komarneni said. "Na-4-mica is not difficult to make and is produced by the same commercial methods used to make other mica-type silicates in industry today."

CRADAs Link Industry with NREL

Four cooperative research and development agreements (CRADAs) have been signed which pair U.S. industry with the

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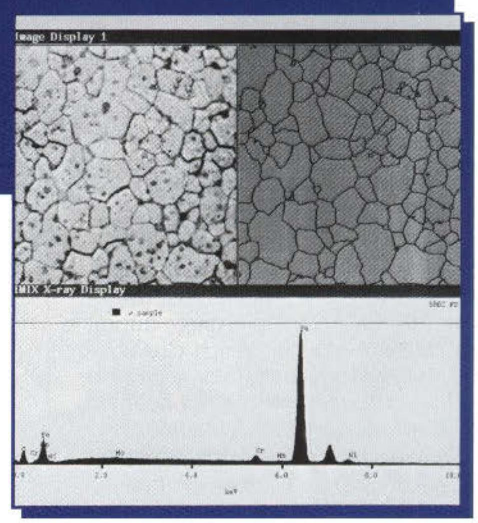

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Department of Energy's (DOE) National Renewable Energy Laboratory (NREL). The four CRADAs involve $36 million of research and development over a period of up to six years and will support efforts such as a novel technique for producing ethanol from the cellulose in waste paper, advanced insulation for new high-performance batteries for electric cars, an economic method of making silicon carbide powders, and a way of using solar energy to reduce electronic component failure rates.

More specifically, the projects include:

- An assessment by Amoco and NREL on the feasibility of converting cellulose to ethanol, as an alternative transportation fuel. The process uses enzymes to break down cellulose into sugars that can be converted to ethanol.
- A project with the U.S. Advanced Battery Consortium, a partnership involving DOE, the nation's big three automakers, the Electric Power Research Institute, and battery companies. Under this CRADA, NREL will develop prototype thermal control systems for different battery technologies based on a high-performance vacuum insulation developed by NREL.
- A solar furnace project for manufacturing silicon carbide powders, conducted by Coors Ceramics Company and NREL, that could greatly reduce the cost of making this material.
- Another solar furnace undertaking with Brush Wellman Inc. and NREL for assembling electronic components. The process would allow researchers to weld metal onto ceramics with pinpoint accuracy.

J.H. Hopps Selected as NSF Materials Research Division Director

John H. Hopps Jr., principal member of the technical staff at the Charles Stark Draper Laboratory in Cambridge, Massachusetts, has been selected to direct the National Science Foundation's (NSF) division of materials research (DMR) of the directorate for mathematical and physical sciences. Hopps will oversee grants in the areas of metals, ceramics, and electronic materials; materials theory; condensed matter physics; solid-state chemistry; and polymers. DMR is responsible for NSF's Materials Research Laboratories.

Hopps' interest has been in quantum-well photonic devices and previously he was instrumental in developing and implementing programs for the application of fault-tolerant control systems to research nuclear reactors and commercial nuclear power systems. He also worked with the Lawrence Livermore Laboratory Mirror

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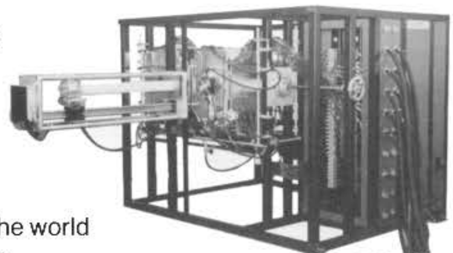

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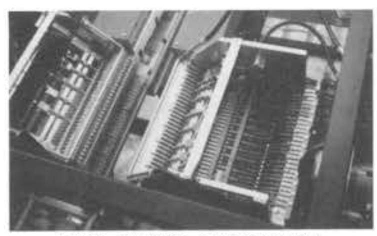

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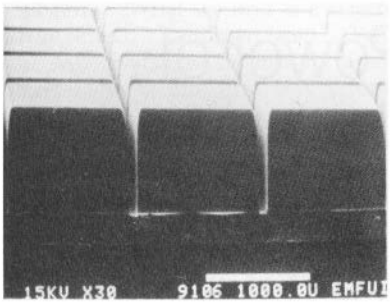

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Hopps has been chief of photonics technology, manager of Draper's Laser Research and Development Facility, and manager of Energy Programs Development at Draper. He was also director of the lab's education programs; he was responsible for student programs having a research component, including student recruitment, evaluation, and facilitation of student research placements. Hopps has been active in the Gifted and Talented Program of the Brookline, Massachusetts School Department, and serves on the Science Advisory Committee of the Cambridge Partnership for Public Education. He holds a PhD degree in physics from Brandeis University.

J.W. Mayer is New Director of ASU Center for Solid State Sciences

James W. Mayer has accepted the position of director of the Center for Solid State Science at Arizona State University. The Center brings together more than 60 researchers in chemistry, physics, geology, and engineering to examine the fundamental structure and properties of matter. Since 1981, Mayer was Cornell University's Francis Norwood Bard Professor of Materials Science and Engineering, specializing in behavior and analysis of surfaces and thin films, primarily in electronics, but stretching broadly into topics such as analysis of art. In 1989, he became director of Cornell's Microscience and Technology Program. Before joining Cornell, he was a professor of electrical engineering at the California Institute of Technology, where he was also a scuba diving instructor and master of student housing. Mayer has also worked at Hughes Aircraft Research Laboratories.

In 1980, Mayer was noted as one of the 1,000 most-cited contemporary scientists from 1965 to 1978, according to the Institute of Scientific Information. He holds seven patents, is co-author of six books, coeditor of three others, and has guided 34 students to doctoral degrees. He received a teaching award from Cornell's College of Engineering in 1990, an honorary doctor of science degree from the State University of New York, Albany, in 1988, and a silver medal from Italy's University of Catania in 1986. In 1981, he received the Von Hippel Award from the Materials Research Society.

He is a member of the National Academy of Engineering and the Böhmische Physical Society, and a fellow of the American Physical Society and the Institute of Electrical and Electronic Engineers.

Mayer received his PhD degree in physics in 1960 from Purdue University.

George Smith Appointed to Readership in Metallurgy at Oxford

George D.W. Smith, Tutor and Fellow in Metallurgy at Trinity College, Oxford, has been appointed to the George Kelley Readership in Metallurgy in the University. Smith's research work has centered on the development of the experimental methods of field ion microscopy and atom probe microanalysis. Smith received two awards for his work, the Sir George Beilby Medal and Prize—awarded jointly by the Royal Society of Chemistry, the Society for Chemical Industry, and the Institute of Metals—and the Rosenhain Medal and Prize, awarded by the Institute of Metals.

Smith, and Oxford colleague Alfred Cerezo, recently developed the position-sensitive atom probe (POSAP), which permits three-dimensional reconstruction of the atomic chemistry of solids. The instrument has a wide range of potential applications, including the study of solid-state phase transformations, and the study of surface chemical reactions.

R.M. German Receives Engineering Award

Randall M. German, professor of engineering at Pennsylvania State University, has been given San Jose State University's Engineering Award of Distinction in recognition of his professional achievement and service. He graduated magna cum laude in 1968 from San Jose State in materials science and engineering. German is the Brush Chair in Materials with the Engineering Science and Mechanics Department at Penn State and was previously the Robert W. Hunt Professor at Rensselaer Polytechnic Institute. He received his PhD degree in materials science from the University of California–Davis.

German's research and teaching focus is on particulate materials processing. He heads Penn State's facility for academic research in powder processing which, along with companion labs on campus, provides significant research capability in powder processing.